

**REPORT DOCUMENTATION PAGE**

AFRL-SR-BL-TR-98-

1/88

Public reporting burden for this collection of information is estimated to average 1 hour per response, including the data needed, and completing and reviewing the collection of information. Send comments including suggestions for reducing this burden, to Washington Headquarters Services, Directorate for Information Operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302, and to the Office of Management and Budget, Paperwork Reduction Project (9700-104).

0317

non, gathering and  
of information,  
in 1984, Arlington,

1. AGENCY USE ONLY (Leave Blank)

2. REPORT DATE

4/7/98

3. REPORT TYPE AND DATES COVERED

Phase I Final Report; 8/15/97-2/14/98

4. TITLE AND SUBTITLE

Nonlinear Optical Polymer Thin Film for the Inspection  
of Sub-Micrometer Electronic Circuits

5. FUNDING NUMBERS

Contract No. F49620-97-0059

6. AUTHOR(S)

Pajo Vujkovic-Cvijin

STTR/TS

65502F

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)

Los Gatos Research  
67 E. Evelyn Ave., Suite 3  
Mountain View, CA 94041

8. PERFORMING ORGANIZATION  
REPORT NUMBER

AF STTR Phase I Final Report

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)

AFOSR/NL  
Directorate of Physics and Electronics  
110 Duncan Ave., Room B115  
Bolling AFB DC 20332-8050

10. SPONSORING/MONITORING  
AGENCY REPORT NUMBER

11. SUPPLEMENTARY NOTES

19980421 057

12a. DISTRIBUTION/AVAILABILITY STATEMENT

Approved for public release;  
distribution unlimited.

12b. DISTRIBUTION CODE

13. ABSTRACT (Maximum 200 words)

Nonlinear optical properties of thin-film polymers are used, together with high spatial resolution of near-field optical microscopy, to create a novel diagnostic instrument for testing VLSI and ULSI integrated circuits (ICs). The instrument performs noninvasive measurements of the electric field generated by sub-micrometer size devices on ICs. Sub-micrometer spatial resolution, and sub-picosecond temporal resolution are both feasible. The instrument is capable of sensing extremely large bandwidth signals from a device within an IC, as well as generating a spatially distributed map of the electric field radiated by the circuit. The instrument is expected to fill a critical need in the areas of circuit design, reliability studies of integrated circuits, multi-chip modules and flat panel displays, and in production quality control

DTIC QUALITY INSPECTED 2

14. SUBJECT TERMS

integrated circuit diagnostics, reliability physics, failure analysis,  
nonlinear optics, polymers, near-field optical microscopy

15. NUMBER OF PAGES

14

16. PRICE CODE

17. SECURITY CLASSIFICATION  
OF REPORT18. SECURITY CLASSIFICATION  
OF THIS PAGE19. SECURITY CLASSIFICATION  
OF ABSTRACT

20. LIMITATION OF ABSTRACT

u

u

u

uL

## 1. Introduction

Los Gatos Research and Washington State University have demonstrated, in the Phase I of this project, a novel optical diagnostic technique for microelectronic integrated circuits (ICs), which combines recent advances in nonlinear optics of polymer thin films, with high spatial resolution of scanning optical microscopy.

Molecular/polymeric systems developed originally for electro-optic and all-optical modulation devices have achieved some of the most remarkable nonlinear optical responses known so far. In principle, when an optical electric field couples with the polarizability of such highly nonlinear molecular system, it strongly affects the molecular electronic structure, affecting not only absorption and refractive index of the material, but also parametric coupling of the optical fields present in the film. These phenomena can be largely affected, even induced, by an external electric field generated by e.g., an electric circuit in close proximity to the film. Consequently, optical phenomena taking place in the nonlinear thin film, are controlled by the electric field produced by the circuit, thereby creating a way for electric field measurement. The possibility exists to create a three-dimensional image of the electric field around the device, which can reveal either its structure or its operation, or both. This information can be used efficiently for reliability/failure analysis studies of ICs. The technique proposed here, which combines optical near-field microscopy with ultrafast nonlinear optical probing, allows mapping of the electric field at the surface of an electronic circuit with the resolution of the order of 100 nm spatially and 100 fs temporally. In another way, the diagnostic instrument described here can be viewed as a "contactless nano-oscilloscope", which can capture waveforms (with ultra-large bandwidth) at desired points on an IC, without making physical contact with the circuit.

The development of the IC diagnostic instrument described above comes at the time when new developments in the semiconductor industry evolve around the following two goals: (1) reducing the device dimensions, and (2) increasing the electric bandwidth (switching speed) of the circuit. At the experimental stage, devices are being fabricated with dimensions smaller than 100 nm, and with switching speeds of less than 10 psec. Such devices are very difficult to characterize with existing instrumentation, whose both spatial resolution and signal bandwidth are limited. Even for applications requiring substantially lower speeds and larger device sizes, there is currently a lack of truly practical techniques to probe an integrated circuit noninvasively, quickly, and at low cost.

Efficient failure analysis and reliability testing is crucial for the ability to develop and manufacture highly reliable, high-yield Very Large Scale Integration (VLSI) and Ultra Large Scale Integration (ULSI) integrated circuits. As both the density of integration and complexity of circuits increase, it is becoming increasingly important to develop new diagnostic techniques which keep up with the pace at which IC feature sizes decrease and packaging density increases, while the reliability requirements increase or, at best, stay the same. The techniques of failure analysis (FA) which have the potential to eventually win broad acceptance in the industry have to be simple to use, fast,

noninvasive, more sensitive than the existing techniques, and should provide spatial resolution and bandwidth appropriate for the size of VLSI/ULSI IC features.

In this proposal, we describe a technique, which relies on the cutting-edge research results in nonlinear optics of organic materials (and advances these further), to address a pressing need in the semiconductor industry described above. If successful, the technique described here will find applications in IC design validation, IC reliability studies, and in quality control. Because of the importance of reliability issues in semiconductor manufacturing, the technology that improves on this crucial feature inevitably draws substantial commercial interest. The manufacturers of ICs, which use such technology, will be able to quickly develop a substantial market advantage relative to those which do not. Consequently, it is conceivable that the global market advantage resulting from technological advancements in this field might affect large segments of the entire national semiconductor industry.

It should be pointed out that the approach proposed here offers other unique advantages, relative to existing diagnostic techniques. First, our technique is completely noninvasive. The circuit under investigation is not connected by any macroscopic connector to the test equipment, making it possible to perform a truly "in vivo" diagnostic of a normally operating (or malfunctioning) integrated circuit. Second, unlike present-day SEM-based techniques, our technique does not require a vacuum system, and additionally, unlike the electron beam and ion beam probing, it does not require sources of ionizing radiation. We believe that these advantages will prove highly valuable for the development of a commercial IC diagnostic instrument.

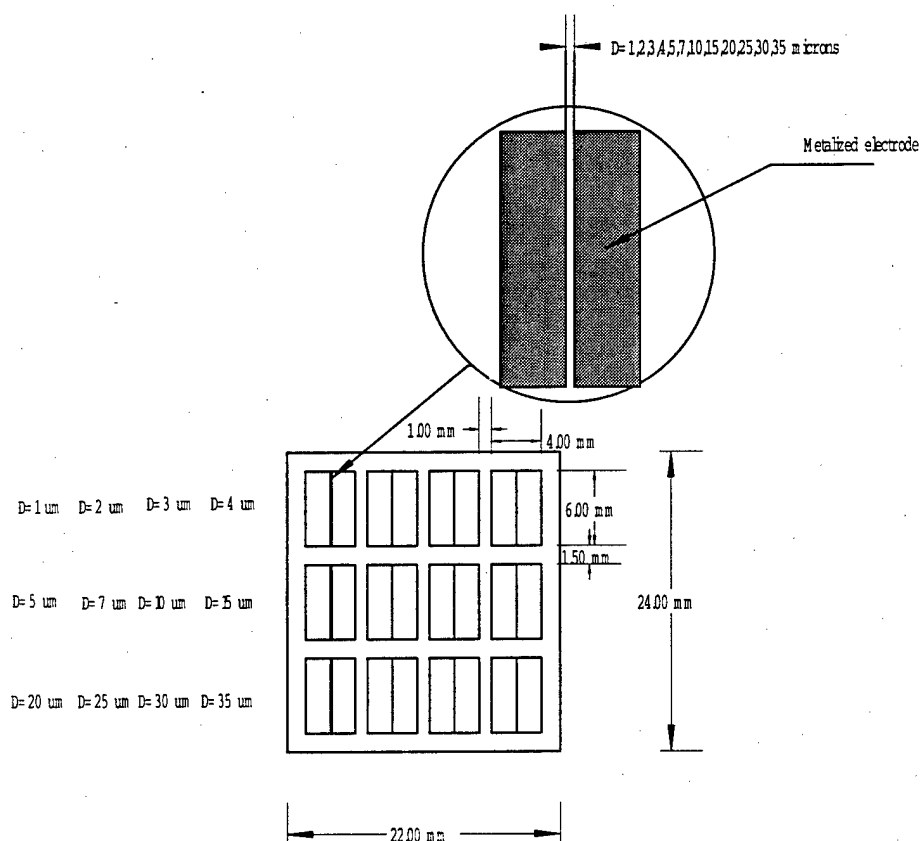
In addition to the opportunity to develop a device with a large applications potential, we intend to advance the scientific and research issues related to nonlinear optics of polymer thin films. We will address the question of nonlinear optical response enhancement by the use of a low glass transition temperature polymer, and the question of resonantly enhancing the product of the ground-state dipole moment with the quadratic hyperpolarizability ( $\mu\beta$ ) term, added to the resonantly enhanced second order hyperpolarizability ( $\gamma$ ). We also propose a radically new geometry for the excitation of highly nonlinear polymeric/molecular systems. Operation under single-, two- and three-photon optically resonant regime becomes also possible, and will be investigated. The resulting diagnostic/imaging instrument will not only reveal the structure of the underlying nanoscale electromagnetic source, but might also become a formidable tool to unravel the electronic structure of molecular systems.

## **2. Phase I Results**

### **2.1. Device Topography**

#### **2.1.1. Device Topography Investigated by Scanning Probe Microscopy**

For the experimental work in Phase I, we have fabricated a series of test devices consisting of chromium electrodes deposited on fused silica substrate. The size of the gap between the two electrodes was varied between  $1\mu\text{m}$  and  $35\mu\text{m}$  (Fig.1.). This structure is suitable for the investigation of the electric field generated between the two electrodes, when voltage is applied to the electrodes. We have started off by investigating the topography of our samples. Fig. 2 shows the topographic image obtained by atomic-force-microscope (AFM) imaging of the electrodes from samples on Fig. 1., separated by a gap of  $2\mu\text{m}$ . The AFM used a SiN tip accurately controlled with the resolution up to  $1\text{ nm}$ . The tip was scanned across the surface of the sample, typically over  $5\mu\text{m}$  distance. The experiments were conducted without prior ultrasonic cleaning; as a result some particles remained in the gap between the two electrodes after manufacturing, as seen on Fig. 2. In addition, we see sharp edges of electrodes, particularly the one at right hand side. An enhanced view is obtained by the topographic projection on the bottom of Fig. 2. In addition, the AFM scan showed that the electrodes had a thickness of approximately  $100\text{ nm}$ . Fig.3. shows that the AFM probe used is clearly able to resolve imperfections or defects with lateral dimensions of approximately  $100\text{ nm}$ .



**Fig. 1.** Samples used for the investigation of the dependence of the electric field-induced second harmonic generation (EFISH) effect on the intensity of the electric field generated by the electric circuit.

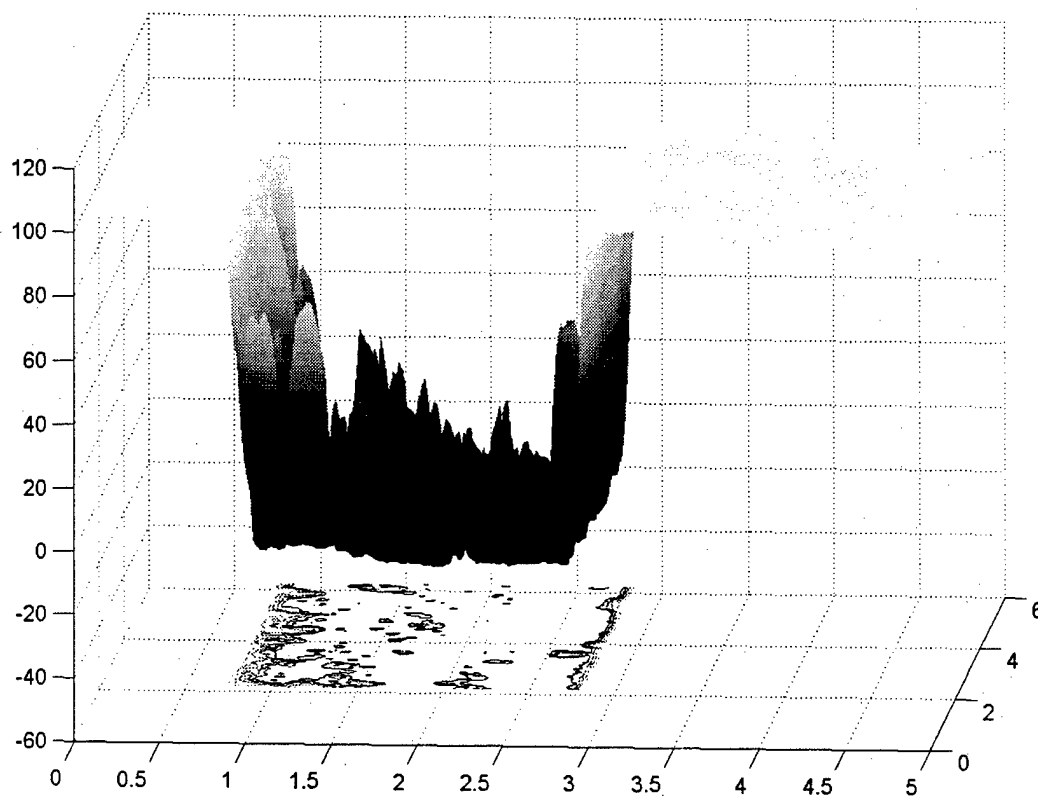


Figure 2. AFM scan of the  $2\mu\text{m}$  gap between two chromium electrodes.

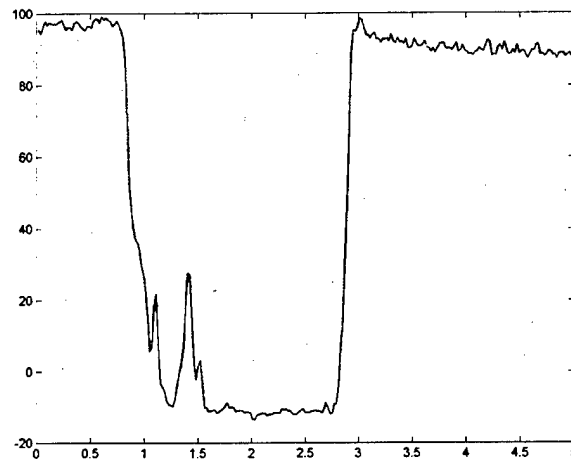


Figure 3. Single-dimensional AFM scan showing a defect in lateral features, approximately 100nm in size. The horizontal axis is in the units of microns and the vertical axis is in the units of nm.

Figs 2.and 3. show sub-micron features inside the gap, arising from imperfections. Additionally, at the edges of electrodes, a rise in the AFM signal shows the resolution of less than 50 nm. The vertical scale has the units of nm while the horizontal and depth axes have the units of  $\mu\text{m}$ .

### ***2.1.2. Device Topography Investigated by Near Field Optical Microscopy***

We have obtained Figs. 4. and 5. by near-field optical microscopy based on the newly developed TIR scheme (see below). Scans were performed over a 10  $\mu\text{m}$  distance. The results presented were obtained with the near field apparatus containing a He:Cd laser operating at 442nm, acquired recently by WSU.

By comparing the AFM and the optical near-field data, we see the remarkable capabilities of the TIR near-field approach. The TIR approach, which we plan to use in Phase II for sub-micron spatial resolution electric field measurements (see below), has thus been demonstrated to measure accurately:

- An electrode gap of 2  $\mu\text{m}$ ;
- A very sharp rise at the electrode edge showing a resolution of approximately 150 nm;
- A depth resolution better than 20 nm and;
- The single defect detected with the AFM is also observed with the TIR microscope with an estimated lateral spatial resolution of 150nm.

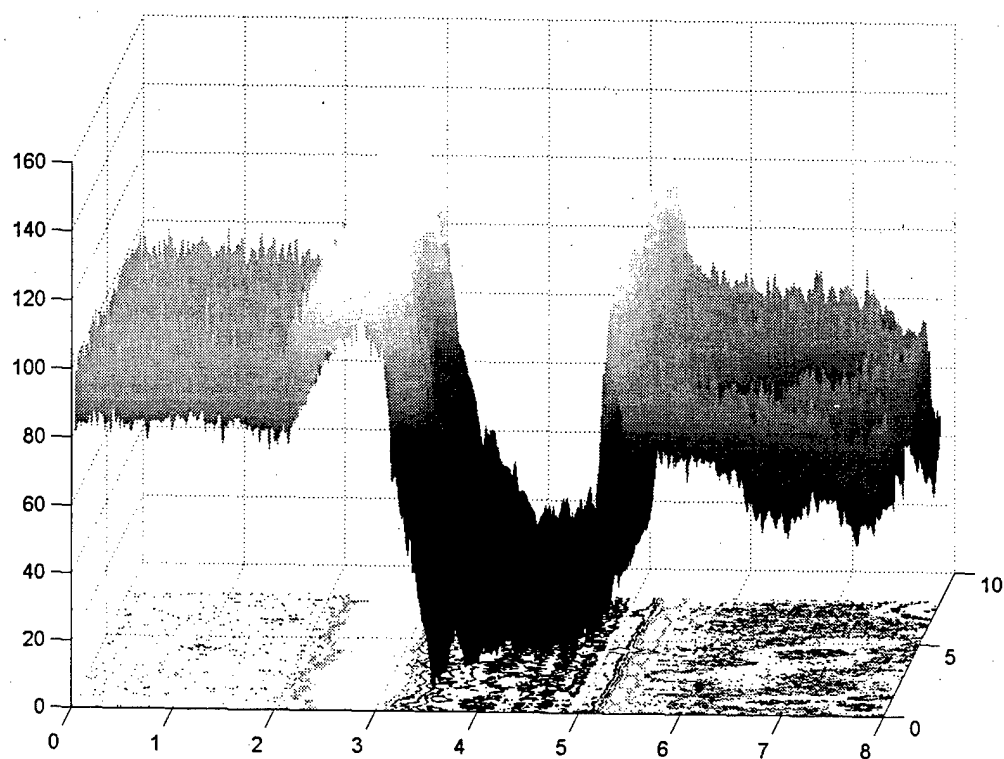


Figure 4. Near-field scan showing a steep rise at the edge of an electrode, in addition to sub-micron features in the gap.

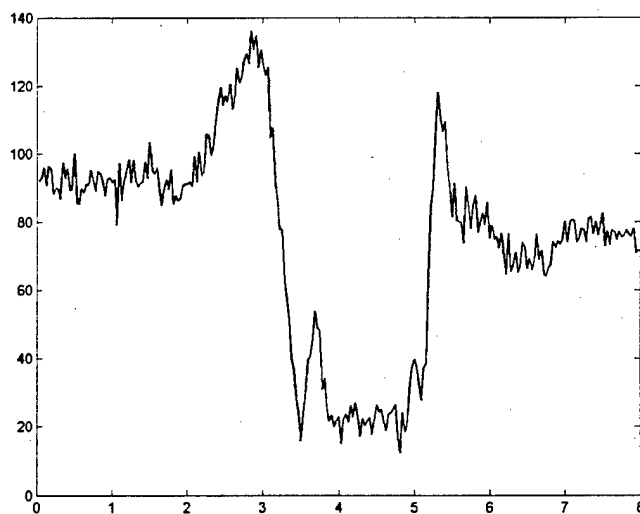


Figure 5. Single near-field scan showing submicron resolution and identifying a single defect within the electrode gap. The horizontal axis is in the units of microns and the vertical axis is in the units of nm.



At the edge of the electrode we observe an overshoot (Figs 4 and 5.). This is a diffraction artifact (light diffraction at a sharp edge), observed also in nonlinear experiments described in the following section.

### ***2.1.3. Device Topography Investigated by Far Field (Conventional) Optical Microscopy***

Simple, conventional, microscopy cannot reach the spatial resolution requirement for the diagnostics of high-density electronic integrated circuits. Although that fact is fairly obvious, we have performed conventional far-field microscopic imaging of our samples, just to complete the comparison of imaging techniques. Standard 50X microscope objective was used. Figs. 6 and 7 show the results. Following conclusions can be drawn from the far-field measurements:

With far-field microscopy, the separation between the electrodes would have been wrongly estimated to be 3  $\mu\text{m}$ ;

The edge of the electrode is convoluted with the optical transfer function of the microscope resulting again in limited resolution of approximately 1  $\mu\text{m}$ ;

Finally, the details obtained due to inner gap imperfections are also masked due to the limited resolution.

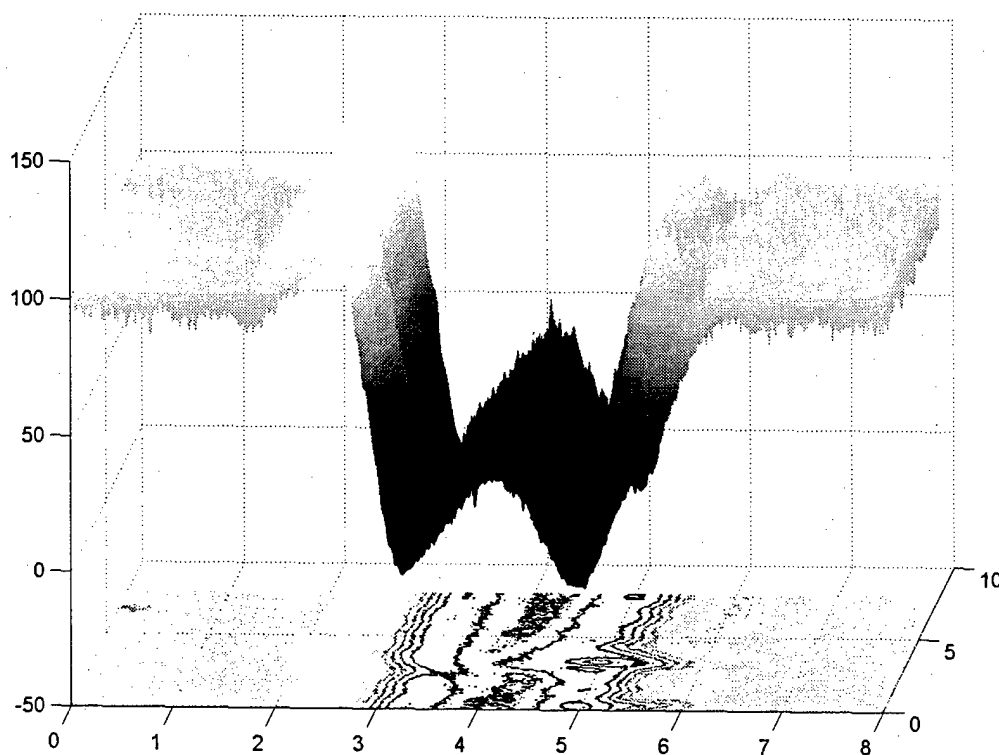


Figure 6. Far-field microscope scan with a 50X objective showing inferior lateral resolution both at the edge of the electrodes and in detecting defects in the gap. Notice in particular the slow rise on the edge of the right electrode and compare it with the AFM and near-field scans.

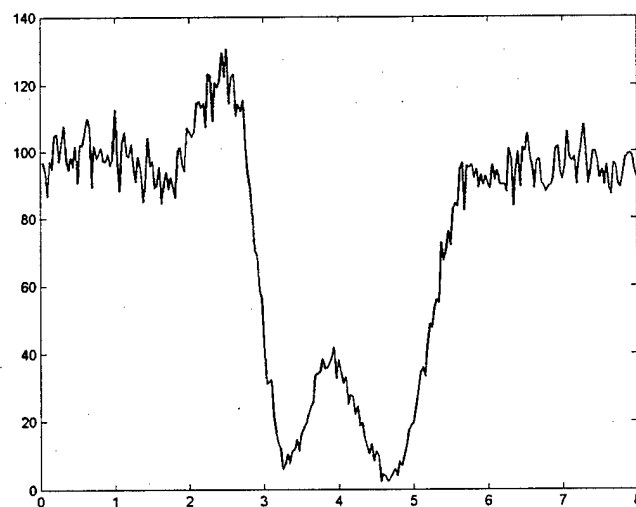


Figure 7. Single scan of the gap with a far-field microscope.

As one would expect, the near-field apparatus provides dramatic spatial resolution improvements over a standard microscope configuration, even with a very large numerical aperture microscope objective. In conclusion, the TIR version of the near-field scanning microscopy provides sufficient spatial resolution (sub-micron) for our needs in VLSI and ULSI circuits.

### ***5.2. Electric Field Measurements by Nonlinear Optical Probe***

Recent experiments have been reported showing that EFISH has a better temporal resolution than electro-optical sampling. The experiments indicate that electric fields can be measured with the resolution higher than 1.95 THz or, equivalently, with better than 1 ps temporal resolution, which can be even further increased, if required. While large bandwidth can be attained relatively easily with a fast laser, pushing the limits in sensitivity and spatial resolution, as required for our application, is much more challenging.

It is quite clear that purely electronic response of a centrosymmetric conjugated molecule should be ultrafast in nature. However, as discussed in the following section, we believe that dipolar push-pull molecular systems are advantageous for this application from the standpoint of enhancing the EFISH signal and thus reducing the detection level of our technique. Additionally, the orientational contribution to the molecular nonlinearity not only will enhance the nonlinear signal, but will allow for the measurement of temporal characteristics on the time scale of the orientational relaxation time, of the order of 100 ps for most molecules of interest in guest-host systems. The latter will result in an additional degree of freedom in choosing the material system: the viscosity of the host polymer medium.

During the Phase I of this project, we have performed electric field measurements by using commercially available charge-transfer molecule DR1. Initial nonlinear optical experiments were performed on samples consisting of two metallic electrodes deposited on fused silica substrates separated by approximately 40  $\mu\text{m}$ , covered by thin polymer films of approximately 1  $\mu\text{m}$  in thickness, deposited by spin coating on top of the electrodes. Deposition thickness was calibrated by monitoring both absorption and fringes, caused by thin film Fabry-Perot effects. With this approach, film thickness could be controlled to better than 100 nm. Films were prepared from solutions of PMMA:DR1. The DR1 concentration was approximately 30% by weight. At room temperature it can be assumed that before applying the DC field, the DR1 molecules were randomly oriented. An ultrafast laser system producing pulses of 100 fsec duration at 200KHz repetition rate and at the wavelength of 930nm, was used to investigate the possibility to detect the second-harmonic signal triggered by the presence of a DC electric field. The wavelength of the laser was chosen to take full advantage of the two photon resonance enhancement of DR1, as discussed in the following section. The results obtained showed that with only 100  $\mu\text{W}$  of average power, a signal could easily be detected with a photomultiplier operated at moderate bias voltages. This indicates that even higher sensitivities can be achieved, or even less optical power may be needed. The laser source

was focused down with a 25X microscope objective, producing a spot of 2 microns in diameter (measured). The applied voltage ranged from 0 to 700 Volts resulting in recorded PMT-signals as high as 500 mV, with an excellent S/N ratio in excess of 100/1. The expected quadratic response on the applied DC field was verified. In addition, when the DC field was turned off, no electric field induced second harmonic (EFISH) signal was observed, indicating no permanent poling of the DR1 molecules. Finally, when focused on an area where the film had been removed, an EFISH signal 1/18000 smaller than the previous signal was observed, indicating that the contribution from the substrate interface and other surface effects is negligible. It is thus easy to anticipate electric field detection sensitivity of a few mV/ $\mu\text{m}$  to be achievable.

The experimental results obtained in our nonlinear optical experiment are presented on Fig. 8. Note the excellent signal to noise ratio, in addition to the 2  $\mu\text{m}$  lateral spatial resolution observable on the edge of the electrode located on the left hand side of the figure. The overshoots of the EFISH signal on electrode edges are partially due to diffraction effects as observed in the linear case, Figs. 6-7. Such effects can easily be removed by taking into account the optical transfer function of the instrument and deconvolving the field pattern numerically. Additional effects such as those due to carrier injection and local field enhancements at the metal surface can also play a role in generating the overshooting artifacts.

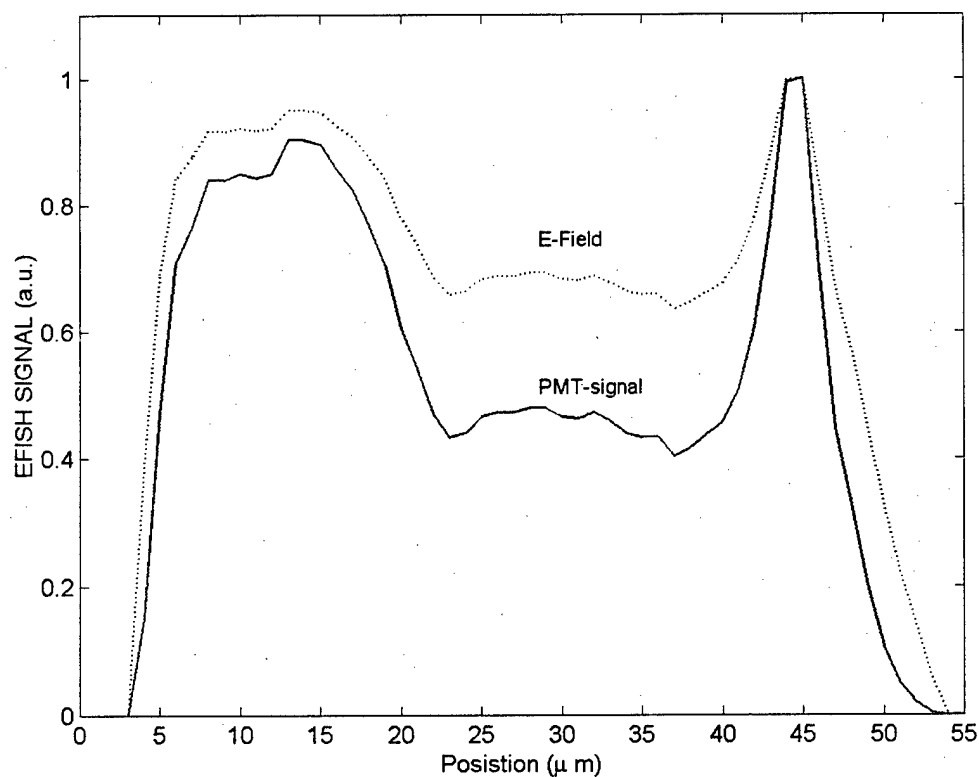


Figure 8. EFISH signal recorded as a function of position for an electrode gap separation of 40  $\mu\text{m}$ .

As described above, we have also separately demonstrated an order of magnitude higher spatial resolution by taking the advantage of the TIR scanning optical microscope in the linear regime. We anticipate with confidence that in Phase II our approach will result in an instrument to optically measure electric fields with spatial resolutions of the order of 100 nm or smaller, with zero background.

### 3. Conclusions

This section directly refers to the objectives and tasks stated in our Phase I proposal and summarizes the results obtained in our Phase I effort.

**Task 1.** We have performed a theoretical analysis based on our understanding of the nonlinear optical response of charge-transfer molecular systems, which has allowed us to identify the appropriate parameters required to investigate EFISH as a probe of electric fields in sub-micron semiconductor devices.

**Task 2.** We have identified Total-Internal-Reflection microscopy as a viable method to conduct nonlinear optical studies of polymer thin layers deposited on the semiconductor surface or on the near-field-microscope tip itself. As a result, a combination AFM/near-field-optical microscope (TIR prototype) has been purchased from Digital Instruments, a leading manufacturer of scanning probe microscopes.

**Task 3.** We have performed femtosecond EFISH experiments on samples consisting of polymer guest-hosts deposited on top of electrodes separated by a gap spacing varying between 1 and 40 microns. We have identified the two-photon excitation spectral region as the preferred spectral region to observe the signal, thereby reducing the minimum electric field that can be observed with our setup.

**Task 3.** Test samples have been manufactured consisting of electrodes separated by gaps of 1-40 microns, covered with PMMA:DR1 thin films deposited by spin coating. The thickness of the spin-coated films has been measured, resulting in reproducible thin films with a thickness accuracy of 50nm.

**Task 4.** Electric fields have been measured with an ultrafast laser source and with previously described test samples exposed to DC fields varying from 1 to 800 V/ $\mu\text{m}$ . EFISH signals were recorded as a function of the optical probe position on the sample, with the spatial resolution of approximately 2  $\mu\text{m}$ .

**Task 5.** An analysis has been performed indicating that fields in the mV/ $\mu\text{m}$  range can be measured with our EFISH near-field approach.

**Task 6.** A marketing survey has been performed. The results are included in this Phase II proposal.